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AN OVERVIEW OF SETTLED DUST ANALYTICAL METHODS AND THEIR RELATIVE EFFECTIVENESS

REFERENCE: Crankshaw, O.S., Perkins, R.L., and Beard, M.E., **"An Overview of Settled Dust Analytical Methods and Their Relative Effectiveness,"** *Advances in Environmental Measurement Methods for Asbestos, ASTM STP 1342,* M.E. Beard, H.L. Rook, Eds., American Society for Testing and Materials, 2000.

ABSTRACT: Methods for sampling and analyzing asbestos in settled dust can be beneficial to document past (and potentially ongoing) episodes of asbestos contamination and to predict potential problems presented by asbestos-containing dust.
Research Triangle Institute conducted an evaluation of several methods for dust collection and analysis, utilizing samples from industrial settings, samples from residential settings, and samples created in a laboratory dust-generation chamber. Sample collection techniques included microvacuuming, wipe sampling, tape sampling, and passive sampling. Analytical methods tested included fiber counting/sizing, fiber mass determination, qualitative analysis, and indirect and direct sample preparation procedures.
The test results help illustrate the advantages and disadvantages of each technique. Each of the methods tested has specific attributes and limitations. Because of the inherent complexity of the methods and the typical variability found in real-world samples, numerous samples of each sample type are recommended, including side-by-side duplicates, representative sampling throughout the target area, and repeat sampling to determine temporal effects.

KEYWORDS: asbestos, settled dust, analysis, transmission electron microscopy, fibers.

The presence of asbestos in settled dust indicates that asbestos has historically been present in the localized atmosphere as airborne particles, and the possibility exists that this asbestos could be re-entrained in the air by future disturbances and activities. Therefore, capable methods for sampling and analyzing settled dust would be beneficial to document past (and potentially ongoing) episodes of asbestos contamination and

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to predict potential problems presented by asbestos-containing dust.

Over the past several years, Research Triangle Institute (RTI), under contract to the US Environmental Protection Agency (USEPA) has engaged in research to test the efficacy of various methods for the sampling and analysis of asbestos in settled dust. Some of these methods were originally developed at the grass roots level; some eventually ended up being refined and formalized as ASTM methods. The goals of the research were to determine the strengths and weaknesses of the various methods, both in real-world residential and industrial settings, and in an artificial laboratory environment where reproducibility, precision, and accuracy could be tested.

The four methods tested in this study include ASTM Standard Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Structure Number Concentrations (D5755), ASTM Standard Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Mass Concentration (D5756), Draft ASTM Standard Test Method for Passive Sampling and Indirect Analysis of Dust Fall by Transmission Electron Microscopy (D22.07.P010.D03), and Draft ASTM Standard Test Method for Sampling and Direct Analysis of Dust for Asbestos by Transmission Electron Microscopy (D22.07.P009.D01).

Description of Dust Sampling and Analysis

For all of the studies, sampling was performed using a variety of techniques to assess type(s) of asbestos present in the settled dust and to quantify asbestos structures per unit area on dust-covered surfaces. Settled dust sampling techniques included aggressive wipe sampling, vacuum sampling, passive dustfall sampling, and adhesive tape sampling. Table 1 shows an overview of each analytical method.

Microvacuum Sampling

Microvacuum sampling utilized a low-volume (2 liters per minute) sampling pump with a 37-mm diameter mixed-cellulose ester (MCE) filter (0.8 µm pore size) attached to the inlet with a length of clear flexible tubing. Attached to the inlet of the cassette was a 2-cm length of tubing to serve as the vacuum inlet. The pump was turned on and the tubing inlet/cassette assembly was swept over a 10-cm by 10-cm sampling area for a 2-minute period, collecting all visible dust. The cassette was then capped, and the cassette and inlet tube were enclosed in an individual plastic bag for storage. A separate cassette and inlet tube were used for each sample. Cassettes and inlet tubes were not reused.

Passive Sampling

Passive sampling involves placing a portable substrate in the path of falling dust. Various media can be used for passive sampling; these studies employed petrislides, which are small round dishes on a rectangular substrate, with a tight-fitting lid. The round

sampling portion of the petrislide represents a 17-cm^2 sampling area. Petrislides, with lids removed, were placed on the substrate in order to collect all dust falling on that location. Following the sampling period, each sampler was removed from the location, and the sampler lid was replaced.

TABLE 1-- *Overview of sampling, preparation, and analytical procedures for settled dust methods*

	Sample Collection	Sample Preparation	Sample Analysis	Reporting of Results
Microvacuum	37 mm MCE filter in cassette with inlet vacuum tube connected to low-vol pump	Suspension of dust and filtering of aliquots followed by AHERA direct preparation	Standard AHERA analysis	Asbestos structures/cm^2
Microvacuum Gravimetric	37 mm MCE filter in cassette with inlet vacuum tube connected to low-vol pump	Weighing of dust and filtering of aliquots followed by AHERA direct preparation	Measurement of each structure and calculation of mass	Asbestos mass/cm^2, percent asbestos in dust by mass
Passive	Open face passive collector, e.g. petrislide or metal film canister	Suspension of dust and filtering of aliquots followed by AHERA direct preparation	Standard AHERA analysis	Asbestos structures/cm^2
Wipe	Ashless cellulose filter dampened with water wiped over surface	Ashing of filter; suspension of dust and filtering of aliquots followed by AHERA direct preparation	Standard AHERA analysis	Asbestos structures/cm^2
Direct Tape Lift	Post-it, commercial tape lift, or others applied to surface and removed	Carbon coat and mount for SEM observation or dissolve sampler and mount for TEM analysis	Examination of fiber/matrix relationship; type of asbestos and other materials; visual estimation	Type of asbestos, visual estimate of quantity, qualification of matrix materials

Wipe Sampling

Wipe sampling used ashless cellulose filters dampened with distilled water to collect the dust on the substrate. Previous studies (Crankshaw et al. 1994) have shown that the use of other wipes or organic filter media results in poor sample preparations because of the presence of residue from the ashing process, which obscures the fibers during analysis. After dampening the cellulose filter with water, the filter was wiped over a 10-cm by 10-cm area of the substrate, collecting all visible dust. The filter was then placed in a glass vial for storage.

Direct Tape Sampling

Direct tape sampling involves removing the dust from the substrate using a sticky sheet of material; the intention is to preserve the dust particle spatial relationships and individual particle structural integrity. Two materials were employed for tape sampling: a commercially available tape sampler and Post-it® notes. The commercial tape lift consists of a dried adhesive applied to a plastic sheet. The adhesive was activated by application of an aqueous solution. The sticky side of the sheet was applied to the dust surface and then peeled off. The sample was then placed in a plastic box (dust side up) for storage. Post-it® note samples were collected in the same manner, except that they required no wetting.

Sample Preparation

Microvacuum structure count samples, microvacuum gravimetric samples, passive samples, and proprietary tape lift samples were all prepared for TEM examination by the appropriate methodology. Tape lift samples and Post-it® note samples were also prepared for scanning electron microscope (SEM) examination.

Microvacuum Samples

The microvacuum samples were prepared for analysis and analyzed by the current ASTM draft settled dust protocol. This involves rinsing the particulate material from the cassette, redepositing an aliquot of the suspension on a secondary MCE filter, and preparing the second filter by the standard Asbestos Hazard Emergency Response Act (AHERA) direct preparation technique.

Microvacuum Gravimetric Samples

Sample preparation began with the desiccation and taring of a blank MCE filter. The sample cassette was then rinsed and the suspension was filtered through the tared MCE filter. This filter was dried, weighed, and then ashed in a muffle furnace at 480 °C for 12 hours. The ash was then rinsed into a laboratory bottle, and the suspension was ultrasonicated for 15 minutes. Aliquots were then filtered onto MCE filters and prepared for TEM examination as described above.

Passive Samples

The passive samples collected on petrislides were prepared by sequential rinsing of the material in the petrislide to create an aqueous suspension of the total material. Aliquots of each suspension were then deposited onto 0.45 μm pore size MCE filters for subsequent direct preparation.

Wipe Samples

The wipe samples were ashed at 480°C for approximately 12 hours to remove all organic material. The residue from each sample was then dispersed by ultrasonication in distilled water. Aliquots of the suspensions were redeposited onto 0.45 μm pore size MCE filters, which were then prepared by the standard AHERA direct preparation technique.

Direct Tape Lift Samples

The commercial tape lift samples were prepared for TEM and SEM examination, while the Post-it® note samples were prepared only for SEM examination. TEM preparation of the tape lift samples involved coating the dust side of the sample with a carbon layer in a vacuum evaporator and then excising portions of the sample for placement on a copper TEM grid. SEM preparation of both the commercial tape lift samples and the Post-it® note samples involved excising a portion of the sample and mounting it, dust side up, on an aluminum SEM stub using conductive paint. The entire sample was then carbon-coated in a vacuum evaporator for sample conductivity.

Sample Analysis

The samples were analyzed by TEM examination; the direct tape lift samples were also analyzed by SEM examination. For the microvacuum structure count samples, the passive samples, and the wipe samples, the goal was to determine the number of structures per unit area. For the gravimetric samples, the goal was to determine the mass of asbestos per unit area and the mass percent of asbestos in the dust. For the direct tape lift samples, the goal was to determine the number of structures per unit area, the relationship of the asbestos to other materials in the sample, and the structural appearance of the asbestos in the sample.

Microvacuum Structure Count, Passive, and Wipe Samples

The microvacuum structure count samples, the passive samples, and the wipe samples were all analyzed by TEM examination using standard AHERA TEM counting rules. This process typically involves noting each asbestos fiber, bundle, matrix, or cluster observed, including classification of structure type and identification. The number of asbestos structures per TEM grid opening were totaled, and the results were calculated incorporating dilution factor, effective filter area, area sampled, aliquot filtered, number of

grid openings analyzed, and grid opening area.

Microvacuum Gravimetric Samples

Microvacuum gravimetric samples were analyzed by measuring the length and diameter of each asbestos fiber or bundle. In order to optimize the representativeness of the analysis, the analysis continued until the mass of the largest bundle in 50 grid openings comprised no more than 10% of the total mass of all structures observed or until the total number of grid openings analyzed was either 100 or [200 000/selected magnification], whichever was smaller.

The size measurements were recorded, and a mass for each fiber or bundle was determined, incorporating the density of chrysotile. A typical analysis consisted of recording the dimensions of 300 to 500 asbestos fibers and bundles and then determining the total asbestos mass for the sample. The total mass for each sample and the mass per unit area were calculated incorporating dilution factor, effective filter area, area sampled, aliquot filtered, number of grid openings analyzed, and grid opening area.

Direct Tape Lift Samples

Only the commercial tape lift samples were analyzed by TEM. The intention of the analysis was to determine the number of structures per unit area on the TEM grid. However, the loading of the samples was so high that it prevented any quantitative analysis due to a method requirement that the grid openings be less than 25% covered with particulate material. Qualitative analysis was done, however, to determine the presence of asbestos, to identify other materials present, and to characterize the structural condition of the asbestos. The commercial tape lift samples and the Post-it® note samples were both analyzed by SEM to determine the structural relationships of the asbestos and the other materials present.

Industrial Study Site

In order to evaluate dust sampling and analytical techniques in an industrial environment, a tire-brake repair shop was chosen as a study site. This location would be expected to generate airborne asbestos because of the long-time use of asbestos in brake linings and the common practice of using jets of compressed air to remove accumulated dust from brake assemblies. No asbestos-containing materials (ACMs) were found in the building, as determined by polarized light microscopy (PLM) analysis of potential asbestos-containing materials. The business had been in operation for about five years at the time of the study. Fortunately for this study, their "housekeeping", or lack thereof, assured an ample supply of dust-covered surfaces. To the best of the employees' memories, the dust had been accumulating since the opening day of business.

Analytical Results

The brake shop represented an industrial environment with the typical combination of airborne dust, oil mist, etc., along with a known source of asbestos dust. All results are summarized in Table 2.

TABLE 2-- *Results of analysis for asbestos in settled dust in an industrial setting*

	Microvacuum Structures/cm^2	Wipe Structures/cm^2	Passive Structures/cm^2	Passive Structures/cm^2 per day
Location 1	120	1 300 000	41 000	1 500
Location 1 duplicate	*	1 400 000	*	*
Location 2	230	1 600 000	89 000	3 200
Location 2 duplicate	*	1 700 000	*	*
Location 3	810	7 400 000	75 000	2 700
Location 3 duplicate	*	6 000 000	*	*
Location 4	*	*	55 000	2 000
Location 5	*	*	62 000	2 100

*samples not collected in these locations

Microvacuum samples--The microvacuum samples all showed low levels of asbestos concentration (below 1,000 structures/cm^2).

Wipe samples--The wipe samples had concentrations ranging from 1.3 to 7.4 million structures/cm^2. Of additional note is the high level of agreement between the three paired samples at each sampling location.

Passive samples--Following one month of sampling, all passive samples showed measurable amounts of asbestos, ranging from 41 000 to 89 000 structures/cm^2. The concentration of asbestos on the passive samples multiplied by the 60 months the shop had been in operation is roughly equivalent to the results from the wipe samples.

Air Samples--Both the inside and outside air samples had no asbestos fibers detected (sampled and analyzed using AHERA procedures for area samples).

Discussion

The low level of asbestos in the commercial site microvacuum samples was notable

since asbestos was found in much higher concentrations in the passive samples. This apparent discrepancy can be attributed to two potential causes: (1) Asbestos settling in the sampling areas may not remain in place; it may later relocate to lower level surfaces or be carried outside the building, and (2) Other substances are also being deposited on the surface, such as oil mist, dust, etc. (not unexpected for an automotive garage) which may cause the asbestos dust to bind to the surface to a degree that it cannot be removed by the light suction of the microvacuum sampling device.

The high level of asbestos in the wipe samples may be attributable to a more complete sampling of the various layers of accumulated material on the substrates in the brake shop.

The presence of asbestos in the passive samples indicates that asbestos is continuously and measurably accumulating on the surfaces at the brake shop. In addition, because the intersample concentration variability was low, the accumulation appears to be rather even throughout the shop. These passive sample results appear to be more relevant to the evaluation of this environment than microvac samples; they demonstrate the dynamic aspect of dust accumulation and serve as an indicator of potential hazard from airborne asbestos dust.

Residential Study Sites

In order to evaluate dust sampling and analytical techniques in a residential environment, two residential study sites were chosen. The first residence, denoted Residence One, contains a known source of asbestos fibers, and the second residence, denoted Residence Two, contains no known asbestos fiber sources.

Residence One was built in 1927; the basement contains a boiler and pipes which are wrapped with asbestos-containing plaster and paper. The asbestos wrap was encapsulated in 1993 with canvas/adhesive, though no overall cleanup of accumulated basement dust was done. Because asbestos was previously identified in this dust, the home has a known source of asbestos fibers for redistribution and accumulation in settled dust.

Residence Two was built in 1970; no ACMs were found in the residence, as determined by PLM analysis of building materials. Theoretically, any asbestos found in the settled dust in this home would have come from outside sources or from items brought into the home subsequent to construction.

Residence One Analytical Results

Residence One represented the location with a friable asbestos source. Of particular concern was the degree to which the asbestos had dispersed throughout the residence, including the upstairs living areas. All analytical results are summarized in Table 3.

TABLE 3-- *Results of analysis for asbestos in settled dust in residential settings*

	Microvacuum Structures/cm^2	Wipe Structures/cm^2	Passive Structures/cm^2	Passive Structures/cm^2 per day
Residence One				
Basement 1	62 000	210 000	77 000	1 700
Basement 1 duplicate	33 000	140 000	*	*
Basement 2	6 800 000	7 000 000	150 000	3 300
Basement 3	350 000	600 000	42 000	910
Basement 4	120 000	310 000	14 000	300
Basement 5	*	*	280 000	6 100
Upstairs 1	7 100	5 300	11 000	240
Upstairs 2	11 000	4 900	7 000	150
Upstairs 3	240	1 800	13 000	280
Residence Two				
Location 1	16 000	23 000	650	6.9
Location 1 duplicate	14 000	9 800	*	*
Location 2	6 700	3 600	330	3.5
Location 3	440	890	330	3.5
Location 4	*	*	<330	<3.5
Location 5	*	*	<330	<3.5
Location 6	*	*	650	6.9

*samples not collected at these locations

Microvacuum samples--The five microvac samples collected in the basement showed levels of asbestos ranging from 33 000 to 6 800 000 structures/cm^2. Two microvac samples taken side-by-side on the duct surface gave results of 62 000 and 33 000 s/cm^2, respectively. The three microvac samples collected in the upstairs occupied portion of the house showed levels of asbestos ranging from 240 (at the detection limit) to

11 000 s/cm^2.

Wipe samples—The five wipe samples collected in the basement showed levels of asbestos ranging from 140 000 to 7 000 000 s/cm^2. All basement wipe samples were taken at side-by-side locations with respect to the five microvac samples, and had, on average, 2.6 times more asbestos than their microvac counterparts. The two wipe samples taken side-by-side on the duct surface gave results of 210 000 and 140 000 s/cm^2, respectively (compared to 62 000 and 33 000 s/cm^2 for the microvac samples in the same location). The three wipe samples collected upstairs showed levels of asbestos ranging from 1 800 to 5 300 s/cm^2. The wipe samples upstairs were comparable to their microvac counterparts from the side-by-side sampling.

Passive samples--The five passive samples collected in the basement showed levels of asbestos ranging from 7 000 to 280 000 s/cm^2. There appeared to be no pattern regarding level of asbestos and proximity to the boiler (the sample taken on top of the boiler had 77 000 s/cm^2). The three passive samples collected upstairs showed levels of asbestos ranging from 7 000 to 13 000 s/cm^2.

Air samples--The two air samples collected downstairs showed asbestos concentrations of 0.077 and 0.098 structures/cm^3 (s/cm^3), respectively. Both upstairs air samples had concentrations below the detection limit (samples were collected and analyzed using the AHERA methodology for area samples).

Residence Two Analytical Results

Residence Two represented the residence for which there is no known asbestos source inside the home. Any asbestos present would presumably have entered the home after the completion of construction.

Microvacuum samples--The four microvac samples showed asbestos levels ranging from 440 to 16 000 s/cm^2 (Table 3). The two samples taken side-by-side had concentrations of 16 000 and 14 000 s/cm^2, respectively.

Wipe samples--The four wipe samples showed asbestos levels ranging from 890 to 23 000 s/cm^2. The two samples taken side-by-side had concentrations of 23 000 and 9 800 s/cm^2, respectively (compared to 16 000 and 14 000 s/cm^2 for the microvac samples in the same location).

Passive samples—The six passive samples taken throughout the residence showed asbestos levels ranging from below the detection limit of 330 to 650 s/cm^2.

Air samples-- No air samples were collected at this site.

Discussion

The presence of substantial asbestos in the basement of Residence One indicates

that the release of asbestos from the boiler and pipes resulted in widespread dissemination of fibers throughout the basement areas. This most likely occurred primarily before the boiler and pipes were encapsulated or during encapsulation activities; however, as the passive samples demonstrate, the dispersion of asbestos fibers is continuing to result in measurable accumulations or redistributions. The effectiveness of the wipe sampling technique was higher than the microvac sampling technique in the basement where concentrations were high. The level of asbestos in the upstairs was substantially lower than in the basement. The lack of accumulation of asbestos upstairs over the 46-day period indicates that asbestos dispersion from the basement to the occupied areas of the residence is minimal to absent.

The levels of asbestos in Residence Two (mean of 9 300 s/cm^2 from the microvac and wipe samples) were comparable to those in the upstairs areas of Residence One. The source of this asbestos is not known; possibilities include items brought into the home subsequent to construction, ambient airborne asbestos, and introduction of asbestos from the clothing of residential occupants. The passive samples had levels 100 times below those from the occupied locations of Residence One (results corrected for sampling time differences), indicating that current accumulation rates are very low for Residence Two.

Where side-by-side sampling was conducted for microvac and wipe samples, the methods show good precision. Wipe sampling appears to be a more effective collection technique than microvac sampling on surfaces containing high asbestos concentrations, but on cleaner surfaces, the microvac and wipe sampling techniques appear to be relatively equivalent.

The highest residential asbestos levels were those from samples taken in close proximity to a known source of asbestos (Residence One). Those concentrations dropped rapidly, however, on samples taken on other floors. While this is partly a function of the ventilation system and air movement in the house, it does demonstrate differential asbestos concentrations in the residence as a probable function of distance.

The concentrations of asbestos in Residence Two indicate that, regardless of construction materials used, asbestos can be found in measureable concentrations even in presumably "clean" locations.

Lab-Created Samples

In order to perform an objective evaluation of dust sampling/analytical methods, a set of standardized, characterized samples was needed. It was determined that real-world settled dust samples were unlikely to provide the necessary uniformity of concentration or the minimization of other variables, including asbestos concentration, matrix type, homogeneity, asbestos fibrous structure size, emission source, emission rate, and dust thickness. For this reason, the dust used for this project was custom-mixed and dispersed in an environmental chamber prior to sampling.

The goal in the creation of the dust for this study was to create a uniform small fiber dimension which would mimic the typical small fibril sizes found with settled dust particles which have traveled in an airstream for a period of time and then settled out onto a horizontal surface. Chrysotile asbestos was combined with the matrix materials by gravimetric addition to yield the following mixtures:

- 0.1% chrysotile in calcium carbonate,
- 1.0% chrysotile in calcium carbonate,
- 10% chrysotile in calcium carbonate, and
- 1.0% chrysotile in vermiculite.

The dust was dispersed in an environmental chamber using a dry powder insufflator attached to a pump operating at 10-liters-per-minute pressure. The dust was allowed to settle for 48 hours in the environmental chamber, at which time the samples were collected. Prior to using the chamber/dust application system for evaluation of the settled dust methods, the homogeneity of the created dust layer was tested gravimetrically and determined to be acceptable.

Analytical Results and Discussion

Eighty-six samples were analyzed, yielding a total of 33 605 chrysotile asbestos structures counted. The median size for the asbestos fibers/bundles was 1.8 μm in length by 0.067 μm in diameter. The mean size was 2.5 μm in length by 0.10 μm in diameter.

Sampling efficiency--Sampling efficiency was determined by subjective means only. The microvacuum sampling method utilized a low-volume pump capable of picking up most loose particles. However, following sampling, there was noticeable material remaining on the sampling surface, indicating incomplete dust collection. The wipe sampling appeared to be more effective, collecting all dust from the sampling surface. The passive samples in effect became the sampling surface, so their efficiency relied on the sample preparation technique. For this experiment and the specific thickness of dust used throughout this study, the direct tape lift sample collection technique was quite inefficient; the samplers only collected the top monolayer of dust, leaving the majority of the material behind.

Microvacuum structure count results--Results from the microvacuum structure count samples indicate that the method adequately tracks the concentration of asbestos in the dust (see Table 4) and that variability is quite low. When the concentration of asbestos was increased ten-fold, as from 0.1% to 1% or from 1% to 10%, the number of structures per area increased proportionately. Also, the variability from sample to sample (intersample variation) within one deposition run should be small, indicating low introduction of variables from sampling and preparation. Low variability from grid opening to grid opening (intrasample variation) indicates low sample preparation variability. Intersample variability was typically less than ±15%. The replicate deposition of 1% chrysotile also produced results very close to the original 1% chrysotile results.

TABLE 4-- *Microvacuum structure count results for lab-created samples*

	Asbestos Structures/cm² x 10^6		
Formulation	**Median Result**	**Intersample Range**	**Inter-Grid Opening Range**
0.1% in $CaCO_3$	1.2	0.80 - 1.4	0.80 - 1.6
1% in $CaCO_3$	7.5	6.2 - 8.4	5.3 - 9.3
1% in $CaCO_3$ (replicate)	7.8	6.3 - 8.8	5.7 - 9.7
3x1% in $CaCO_3$	9.8	9.0 - 11	7.9 - 11
10% in $CaCO_3$	150	140 - 180	120 - 180
1% in Vermiculite	5.6	4.8 - 5.9	4.2 - 7.1

Microvacuum gravimetric results--The gravimetric samples did not perform as well at tracking asbestos concentration (Table 5) and had considerably higher intersample variability (typically ±80%). The asbestos mass percent concentration found in the gravimetric samples did not correspond well with the known concentration of the asbestos dust. Though some results, especially at the higher concentration levels were reasonably close, the intersample variability caused the average result for each concentration to be significantly biased.

TABLE 5-- *Microvacuum gravimetric sample results for lab-created samples*

	ng Asbestos/cm²		
Formulation	**Median Result**	**Intersample Range**	**Percent Asbestos**
0.1% in $CaCO_3$	69	49 - 86	0.0018 - 0.0028
1% in $CaCO_3$	3 300	730 - 7 600	0.032 - 0.26
3x1% in $CaCO_3$	22 000	1 100 - 55 000	0.044 - 2.4
10% in $CaCO_3$	140 000	45 000 - 290 000	1.5 - 13

Most of the variability in the gravimetric results was due solely to the effect of the single large initial structure. Only 3 of the 12 analyses continued until the relative mass of the first large structure diminished to 10%; the others were stopped automatically after 100 grid openings, as specified by the method. An average of 2 000 grid openings would

have been required to reduce the first structure to the 10% level, and in one case 10 000 openings would have been required. This has the effect of proportionately increasing the bias caused by the first large structure, thereby increasing method variability.

Passive results--The passive samples also tracked the asbestos concentration well and showed low variability (Table 6). Intersample variability was typically less than ±15%. The quantitative results of the passive samples were reasonably equivalent to the microvacuum structure count samples. Though the passive sampling was more efficient, these samples represented a smaller collection area, and any loss of fibers due to sample preparation would have been proportionately larger than with the microvacuum structure count samples, which would have balanced out the final concentration to some degree.

TABLE 6-- *Passive sample results for lab-created samples*

	Asbestos Structures/cm² x 10^6		
Formulation	**Median Result (normalized to 7 g dust/application)**	**Intersample Range**	**Inter-Grid Opening Range**
0.1% in $CaCO_3$	0.87	0.79 - 0.98	0.56 - 1.2
1% in $CaCO_3$	7.0	6.0 - 7.8	5.6 - 8.3
3x1% in $CaCO_3$	10	9.7 - 11	9.2 - 11
10% in $CaCO_3$	130	120 - 140	110 - 150
1% in Vermiculite	6.6	5.3 - 7.9	5.0 - 8.4

Wipe results--The wipe samples had the best performance in tracking asbestos concentration (Table 7), with 1.9 million structures/cm² (Ms/cm²) at 0.1%, 17 Ms/cm² at 1%, and 200 Ms/cm² at 10%. Intersample variability was also less than ±15%. In terms of absolute concentration, the wipe samples were the most effective at fiber collection and retention during preparation, as evidenced by their higher concentration of fibers per unit area compared to the microvacuum structure count samples and the passive samples.

Direct tape lift results--As indicated above, the commercial tape lift samples were unable to be analyzed by TEM as specified by the method, which requires appropriate loadings of less than 25%. SEM examination of the two types of tape lift samples (commercial tape lift samples and Post-it® samples) was carried out, and appeared to be a more appropriate method of examination for these sample types. With SEM analysis, identification of the asbestos fibers and other particles by x-ray spectroscopy and fiber morphology was feasible, and structural relationships between the asbestos and non-asbestos particles (as they appeared on the tape lift sampler surface) was readily observable.

TABLE 7-- *Wipe sample results for lab-created samples*

	Asbestos Structures/cm^2 x 10^6		
Formulation	**Median Result**	**Intersample Range**	**Inter-Grid Opening Range**
0.1% in $CaCO_3$	1.9	1.6 - 2.1	1.6 - 2.3
1% in $CaCO_3$	17	16 - 18	15 - 19
3x1% in $CaCO_3$	17	16 - 18	16 - 19
10% in $CaCO_3$	200	190 - 200	170 - 230
1% in Vermiculite	7.4	7.3 - 7.5	6.5 - 8.4

Summary and Conclusions

The RTI studies have demonstrated that the settled dust methods have low internal variability, and with the exception of the microvacuum gravimetric method, are quite precise. However, each of the methods tested has specific attributes and limitations. Depending upon the environment, some sampling and analysis techniques for settled dust may have greater utility than others, and assumptions made for one environment may not hold true for another. For instance, where settled dust is very loose and is not currently accumulating, the microvac sampling technique should be more appropriate than the passive sample approach. Where dust is still accumulating, and adhering tightly to the surface as it collects (as appeared to be the case at the industrial site), a combination of passive sampling and wipe sampling would be more appropriate than microvacuum sampling.

Passive collection techniques and active collection techniques (microvac and wipe) are both necessary to determine current asbestos accumulation and historical asbestos accumulation. Microvac samples tend to more accurately reflect potential re-entrainable asbestos, wipe samples tend to more accurately reflect all accumulated asbestos, and passive samples provide a measure of current accumulation rates. Air sampling provides a snapshot in time of airborne fiber levels.

It should be pointed out that real-world samples will be likely to have substantial variability, though under the best circumstances (little or no bias or variability introduced during sample collection or preparation and analysis) the variability found should be attributable to the samples themselves and not to the method or to the personnel collecting or preparing the samples.

A comprehensive, effective approach to settled dust analysis would utilize more

than one method in order to determine historical accumulation, loose versus bound dust, source location, and current accumulation. Because of the inherent complexity of the methods and the typical variability found in real-world samples, numerous samples of each sample type are recommended, including side-by-side duplicates, representative sampling throughout the target area, and repeat sampling to determine temporal effects.

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